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A METHOD FOR DETERMINING LIQUID VOLATILITY
AT AMBIENT TEMPERATURES USING AN
INFRARED GAS ANALYZER (U)

by

C.G. Martin, J.M. McAndless and C.L. Chenier

PCN No. 13E20

July 1982



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ABSTRACT

A method is described for directly measuring liquid volatility at ambient temperatures without the need to measure vapour pressure or use a predictive vapour pressure-temperature relationship. The method is based on the drying of a wetted solid under controlled conditions and relating volatility to the rate of drying. The rate of drying is determined by monitoring changes in vapour concentration over time using an infrared gas analyzer.

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INTRODUCTION

Volatility can be used to quantify and compare the evaporative characteristics of different liquids. This parameter is calculated from vapour pressure data using the Ideal Gas Law.

Several different methods are available for measuring the vapour pressures of liquids. These methods include vacuum distillation, differential thermal analysis (1,2,3), gas chromatography (4,5), thermogravimetry (3,6), flow-through flame ionization (7), effusion and gas saturation. A review of such methods has been published (8).

By measuring the vapour pressure of a liquid at several different temperatures at a constant ambient pressure over a limited range, a relationship between vapour pressure and temperature can be established. Usually, this relationship takes the form:

... 1

 $\log p = AT^{-1} + B$

where p = vapour pressure of liquid (mm Hg)

T = temperature (°K)

A, B = constants. In some cases, B is another function of T.

Equation 1 does not precisely express the behavior of all liquids and generally this type of equation is linear only over a narrow range of temperatures. Therefore, a whole series of semi-empirical equations have been proposed, many of which are modified versions of Equation 1, in order to define the vapour pressure-temperature dependence of given liquids (for a review of these equations, see Reference 9). Once a relationship has been established, the vapour pressure of the liquid (and also its volatility) can be calculated fc any given temperature.

For liquids of low and intermediate volatility, an extrapolation of the vapour pressure-temperature function from data acquired at higher temperatures (e.g., $50 - 200^{\circ}$ C) is usually necessary in order to calculate the vapour pressures of such liquids at ambient temperature (e.g., 20° C). This can produce large errors in the calculated values depending on the accuracy associated with the function constants A and B.

In this Report, a new method for directly measuring liquid volatility under ambient conditions is described. This method is particularly suited for determining the volatilities of liquids which have relatively low vapour pressures or for cases where there is no requirement to derive a predictive vapour pressure-temperature relationship. The method involves drying a wetted solid under a continuous and constant air flow while monitoring changes in vapour concentration in the drying air. A series of liquids of known volatility were applied to a common solid substrate which was then dried under controlled conditions at ambient temperatures in order to establish a predictive relationship between liquid volatility and drying curve area (accumulated vapour dose). A long-path infrared gas analyzer was used to continuously measure vapour concentrations as produced by liquid evaporation under the given conditions.

THEORETICAL CONSIDERATIONS

Figure 1 shows a typical drying curve for a wetted solid and illustrates the different stages in the drying process which occur under constant conditions of air flow, temperature, pressure and humidity. The detailed theory associated with the process of drying is described elsewhere (10, 11).

Under constant conditions, a thoroughly wetted solid initially dries according to the rate at which liquid evaporates from the surface and diffuses into the air stream above the solid. That is, the mechanism for "constant rate" drying is that of evaporation from a liquid surface and the presence of the solid does not affect this mechanism. The diffusional process essentially involves the transfer of molecules from the liquid phase into the air under the influence of a vapour concentration gradient (12). The temperature at which the liquid reservoir is maintained governs both the rate of vapour production (generation rate) and the vapour pressure above the liquid. The diffusional process associated with "constant rate" drying is described (12) according to the equation:

 $G = (2.303 \text{ DMPA})(RTL)^{-1} \log_{10} [P(P-p)^{-1}] \dots 2$

where $G = \text{vapour generation rate } (g \text{ min}^{-1})$

D = molar diffusion constant of vapour into the air stream at temperature T and pressure P (cm² min⁻¹)

M = molecular weight (g mole⁻¹)

A = cross sectional area of diffusion path (cm²)

 $R = gas constant (L atmospheres mole^{-1} °K^{-1})$

T = temperature (°K)

L = length of diffusional path (cm)

P = ambient pressure (atmospheres)

p = vapour pressure (atmospheres)

This equation is similar to that which describes the production of vapour from a diffusion tube containing a reservoir of liquid (12).

For the case of liquid evaporating from the surface of a solid of cross-sectional area A, the length of the diffusional path L is influenced by boundary air layer conditions and air flow rate. Under constant, non-turbulent laminar air flow, L will attain a small and constant value. Thus, under steady state conditions of airflow, temperature, pressure and diffusional path geometry (AL⁻¹), the vapour generation rate is constant and is proportional to the vapour pressure of the liquid.

At a certain point in the drying process, the surface of the solid will no longer be completely covered with liquid and the drying rate will decrease. This "falling rate" drying stage is characterized by two distinct phases. During the first phase, the vapour generation rate decreases in a linear fashion as the surface area covered by the liquid becomes progressively smaller. Eventually, no liquid remains on the surface and the rate of drying is then controlled by liquid/vapour diffusion through the solid matrix to the surface. Under these circumstances, the diffusion rate decreases with decreasing liquid content in the solid. During this second phase, the thickness of the solid becomes an important variable and air flow is less influential. This final drying phase is characterized by a complex, non-linear relationship between drying rate and amount of liquid remaining in the solid.

Under steady-state conditions, changes in the vapour concentration in the air stream will reflect the drying curve illustrated in Figure 1. That is, vapour concentration is given by the following equation:

$$C = GJF^{-1} ... 3$$

where C = vapour concentration (vol/vol or ppm)

 $G = vapour generation rate (g min^{-1})$

J = reciprocal vapour density (L g⁻¹)

 $F = air flow rate (L min^{-1})$

The reciprocal density can be calculated as follows:

$$J = \frac{22.4}{M} \times \frac{T}{273.2} \times \frac{760}{P}$$
 ... 4

where 22.4 = molar gas volume at standard temperature and pressure; and

M, T and P are as previously defined for equation 2 (P in units of mm Hg).

Thus, vapour concentration in the air stream above the solid is proportional to the vapour generation rate (and also vapour pressure) provided the surface of the solid is covered totally or partly with liquid.

APPARATUS AND PROCEDURES

In developing a method for measuring liquid volatility based on the drying process, the following points were considered:

a) volatility should be determined under ambient temperature conditions without the need to measure vapour pressures over a temperature range and employ a predictive vapour pressure-temperature relationship;

- b) the physical properties and molecular weight of a given liquid may be unknown initially thereby precluding a calculation of volatility by means of vapour pressure data and the Ideal Gas Law;
- c) vapour concentration measurements should be carried out only during that part of the drying process where vapour generation rate is governed solely by liquid evaporation from the solid surface.

Figure 2 shows the apparatus used in the present study. The main components consist of a glass chamber into which a wetted solid can be inserted and a detector for continuously monitoring vapour concentration in the air flowing over the solid. The chamber (length = 30 cm, cross-sectional area = 4 cm²) was tapered at one end to a tube of 1.25 cm diameter and equipped at the other end with a \mathbf{T} 29/42 ground glass joint. The tapered end was connected directly by a short length of teflon tubing to the inlet port of a Wilks Miran 1A Infrared Gas Analyzer equipped with a 20 m pathlength gas cell. A charcoal-filled canister was mated via a ground glass joint to the other end of the chamber to filter any traces of organic vapours from the incoming air. Air was drawn through the system at 19.8 \pm 0.5 L min⁻¹ by means of the analyzer air pump.

For most experiments, a glass frit (Kimflow Disc, Fritted, Size 30 F, diameter = 3.0 cm) was used as the substrate for the test liquids. A nylon/cotton 50/50 blend fabric was used in a few cases for comparison purposes. Opposite sides of the frit were shaved slightly with a file until it could be fitted into the chamber at an angle of approximately 45° from the horizontal. The frit was also ground smooth on the surface opposite to the one to which liquid was applied. Care was taken to ensure that each frit had the same overall area (7.0 cm²) after these modifications. Fabric samples which were cut to the same area could also be placed at a 45° orientation in the chamber provided the sample was inserted carefully. These modifications were required as a glass chamber of a larger size to suitably accommodate the samples was not immediately available.

The entire surface of a glass frit or fabric could be thoroughly wetted by applying $100 - 200 \mu L$ of liquid.

Table I (Annex A) lists the ten liquids which were used to establish a predictive relationship for determining volatility at ambient temperatures. The analytical wavelength and published vapour pressure data associated with each of these liquids is also included in the table.

A typical procedure for each experiment was as follows:

a) one frit or one fabric sample was inserted into the chamber near the end equipped with the ground glass joint. The chamber, analyzer and

canister were connected together as shown in Figure 2;

- b) air was drawn through the apparatus for at least 30 minutes to allow the system to equilibrate to ambient conditions. The air temperature was monitored by a YSI Model No. 705 thermistor attached to the inlet of the canister. Generally, ambient temperature was $23 \pm 2^{\circ}$ C and the relative humidity was 35 55% RH during the several months over which experiments were carried out. For individual experiments, the temperature and humidity varied little over the 60 180 minutes normally required for data acquisition;
- c) the analyzer was set at the characteristic infrared absorbance wavelength of the liquid to be tested and a baseline ("zero") reading was established. The absorbance wavelength was chosen previously by filling the gas cell with vapour, recording the infrared spectrum of the vapour over the wavelength range 2.5 14 microns and selecting a wavelength where the sample exhibited strong absorbance. Other analyzer and recorder parameters which were held constant for all experiments are listed in Table II;
- d) the canister was momentarily disconnected from the chamber and the substrate removed;
- e) the substrate was wetted with test liquid, (usually 100 μ L or 150 μ L) by using a Centaur pipette of the appropriate volume;
- f) the substrate was immediately placed in the chamber close to the tapered end using a glass rod with a slotted end to slide the sample. Care was taken to avoid contacting the chamber walls until the sample was in place;
- g) the canister was reconnected to the chamber and absorbance readings were continuously recorded until such time as baseline readings were approached;
- h) the recorded curve was integrated over the time required for absorbance readings to reach a value equal to 0.25 of the initial value (see DISCUSSION).

RESULTS AND DISCUSSION

1. Data Interpretation

Assuming the vapour evolving from the wetted substrate behaves as an ideal gas.

the infrared absorption at a given wavelength is proportional to the vapour concentration according to the well-known Beer-Lambert Law:

$$A_{\lambda} = a_{\lambda}bC$$
 ... 5

where A_{λ} = absorbance at wavelength λ

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 $a\lambda$ = molar infrared absorbtivity coefficient at wavelength λ (m⁻¹)

b = cell path length (20.25 m)

C = vapour concentration (ppm)

Substituting for C and J in Equation 3 and combining with Equation 2 yields an equivalent expression in terms of absorbance:

$$A_{\lambda} = k_1 \log_{10} [P(P-p)^{-1}]$$
 ... 6
where $k_1 = 2.795 \times 10^6 a_{\lambda} DA(LF)^{-1}$

Thus, under steady state conditions of temperature, ambient pressure, diffusion geometry, air flow, etc., the vapour source is characterized by the temperature-dependent terms D, a_{λ} and p. The use of absorbance rather than concentration allows experiments to be performed on both known and unknown vapour sources without having to produce a calibration curve for the infrared analyzer in each case.

Figure 3 shows three typical absorbance versus time curves which are produced when drying the glass frit wetted with liquids of different volatility. These curves have the general shape of the typical drying curve shown in Figure 1 with the exception that the transition point from "constant rate" drying to "falling rate" drying is relatively poorly defined in the present experiments. In addition, there is a concentration "spike" at the start of some experiments. This effect is due to interruption of the airflow through the chamber while the wetted substrate is inserted. Generally, the time over which the initial concentration "spike" returns to level absorbance readings is small compared to the total experiment time.

The ill-defined transition from "constant rate" drying and the presence, in some cases, of concentration "spikes" preluded using only the constant absorbance portion of curves to relate to liquid volatility. Therefore, an empirical approach was taken in which the integrated drying rate (i.e., area under the curves) was used to correlate with liquid volatility. The curves were integrated to that point in time where the absorbance reading

had fallen to 0.25 of the highest value recorded. That is: Curve Area = $\int_{t_0}^{t_A} A_{\lambda} dt$

where t_A = time at which absorbance = 0.25 × highest absorbance reading (t_0). The end point, t_A , was generally on the "falling rate" portion of the curves and occurred before the non-linear portion of the curve associated with the substrate-controlled diffusion process. This choice of time span ensured that the calculated area was related to the situation described by Equation 2 (i.e., constant rate of vapour generation followed by a linear decrease in generation rate corresponding to a decrease in surface area of the liquid reservoir).

In most cases, the highest absorbance reading was also the initial value associated with constant rate drying (for example, see drying curves for anisole or cyclohexanol shown in Figure 3). For vapour sources which produced concentration "spikes", the well-defined spike peak was used to provide the initial (highest) absorbance reading. For these cases, little difference was found (e.g., 1-3%) when comparing intergrated curve areas calculated using initial absorbance values associated with either the spike peak or the start of constant rate drying. This relates to the fact that the small increase in area produced by the spike is offset by a similar "loss" of area when integration is carried to the end-point, tA, which depends on the initial absorbance value chosen.

As indicated in Figure 3, liquids of relatively high volatility such as anisole dry faster (produce less curve area) compared to liquids such as dimethyl sulfoxide which have lower volatility. This is also shown by the data in Table III. The molar absorptivity coefficient, $a\lambda$, for each liquid is also included in this Table to show that changes in the magnitude of the coefficient are not related to the observed changes in drying curve area. The absorptivity coefficients were determined by calibrating the IR analyzer against known vapour concentrations of the appropriate compound under ambient conditions and applying Equation 5.

2. Relationship Between Volatility and Drying Curve Area

a) Glass substrate. Using the procedure described above, three replicate runs were carried out with each of the ten liquids listed in Table I. Glass frits were used as the substrate and these were wetted with either $100 \mu L$ or $150 \mu L$ of the appropriate liquid. Experiments were carried out under steady ambient temperature conditions; in general, a run was rejected if the temperature changed more than $1^{\circ}C$ while data was being accumulated. The results for the experiments using either $100 \mu L$ or $150 \mu L$ liquid volume are summarized in Tables IV and V, respectively.

Figure 4 shows a log-log plot of the data in these two Tables and indicates that a linear relationship exists between volatility and drying curve area over the range of volatilities studied. The function parameters and correlation coefficients are listed in Table VI.

The average drying curve area ratio (i.e., curve areas for 150 μ L of liquid versus curve areas for 100 μ L of liquid) for the ten compounds was found to be 1.49 \pm 0.11. This ratio is in good agreement with the actual liquid volume ratio of 1.50.

b) Fabric substrate. For comparison purposes, a small number of experiments similar to those described above were performed using nylon/cotton fabric as the solid substrate. The fabrics had the same diameter and area as the glass frits but were much thinner. The data for these experiments which involved using 150 μ L of liquid in each case is summarized in Table VII. Also included in this table is a summary of the relative difference produced between drying curve areas for each liquid when going from a glass frit to a fabric substrate.

These results indicate that there is a reasonable correlation between the experiments carried out with the two different substrates and that, under the given conditions, the substrate type does not influence the vapour generation rate, as predicted by Equation 2.

3. General Comments

As described above, the method for determining liquid volatility requires that experimental parameters including air flow rate, analyzer settings, sample geometry, and ambient conditions remain constant and reproducible in order to establish a predictive relationship between volatility and drying curve area. Of prime importance is the requirement to maintain constant temperature since several temperature-dependant factors contribute to the vapour generation rate and ultimately the drying curve area. These factors include the molar diffusion constant, vapour pressure and infrared absorptivity coefficient associated with each liquid. In situations where non-stable ambient temperature conditions prevail, the apparatus could be readily modified to incorporate temperature control for the air stream, chamber and gas cell.

Other factors which can affect the vapour generation rate and must be closely controlled are the rate of air flow and boundary air layer conditions near the wetted substrate. The latter conditions affect the vapour diffusion path length which can vary considerably under turbulent air flow.

From the above discussion, it is evident that any advanced version of the current apparatus should incorporate accurate temperature and air flow control as well as means to ensure that air movement over the wetted substrate is such that the diffusional path length remains constant. In addition, a means should be investigated of introducing the sample into the chamber so as to prevent initial vapour concentration "spikes". A

chamber which will accommodate different-sized substrates without requiring any modification to the substrate itself would be desirable. Other vapour detectors such as flow-through flame-ionization or flame-photometric devices may prove to be useful alternatives to the infrared analyzer used in this study.

The ability to determine liquid volatility at ambient temperature without the need to use or produce a vapour pressure-temperature function is one of the advantages of this method. If the liquid is unknown, the method can still be applied provided the associated vapour behaves as an ideal gas (usually valid over the relatively low vapour pressures and concentrations involved).

With appropriate precautions taken, the apparatus could readily be used to determine the volatility of hazardous and toxic liquids. The analytical wavelength of such compounds can be determined by examining the infrared spectrum of the vapour while the vapour is present in the analyzer gas cell.

Preliminary experiments have been carried out on liquids with volatilities which are beyond the range of those in the present study. Liquids with volatilities in the range 50 - 1500 mg m⁻³ generally follow the predictive equation but in practical terms the time required to complete the runs becomes inconveniently long (e.g., greater than 5 hours). Very volatile liquids dry too rapidly under the experimental conditions to permit curve areas to be measured accurately.

CONCLUSIONS

- 1. Under steady state drying conditions, the rate at which a wetted solid dries can be related via a linear relationship to the liquid volatility.
- 2. The relationship between liquid volatility and drying rate is not affected by the nature of the solid substrate provided the solid surface is completely or partly covered with liquid.
- 3. Changes in infrared absorbance rather than vapour concentration can be used to establish drying rate curves for subsequent integration and correlation with liquid volatilities. This presumes that large differences do not exist between molar infrared absorptivity coefficients for the liquids used to establish the relationship between volatility and drying rate.
- 4. The method can be used to determine volatilities of liquids without requiring vapour pressure data or other associated physical constants.

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ANNEX A

LIST OF TABLES

TABLE I

Liquids Used To Develop Volatility-Drying Rate Relationship

Liquid	Designation	Analytical Wavelength	Vapour Pressure ^a	Volatility ²	Reference	
2140.0	20018-1000	(microns)	(mm Hg)	(mg m ⁻³)	Activities	
triethyl phosphate	TEP	9.45	0.218	2,140	13	
dimethyl sulfoxide	DMSO	9.05	0.602	2,520	14	
acetophenone	ACP	7.90	0.500	3,230	15	
dimethyl malonate	DMM	8.60	0.506	3,592	15	
N-ethyl-methyl carbamate	EMC	8.65	0.998	5,374	15	
cyclohexanol	CHA	9.30	1.635	8,790	15	
n-amyl alcohol	NAA	9.40	2.660	12,600	15	
iso-amyl alcohol	IAA	9.40	3.715	17,600	15	
anisole	ANS	9.45	3.954	23,000	15	
n-butanol	BTA	9.60	7.252	28,900	15	

a at $T = 25^{\circ}C$

TABLE II

IR Gas Analyzer and Recorder Settings

Parameter	Setting
cell pathlength	20.25 m
air flow	$19.8 \pm 0.5 \text{ L min}^{-1}$
slit width	l mm
absorbance range	0.025 - 1.0
recorder span	0 - 1V full scale
wavelength	absorbance maximum for each liquid (see Table I)
vapour detection limits	approximately 0.1 ppm for liquids tested

TABLE III

Comparison of Integrated Curve Area, Volatility and Molar
Absorptivity Coefficient for Three Representative Liquids

Liquid	Curve Areaab	Volatility @ 25°C	Molar Absorptivity a)
	(A.U. minutes)	(mg m ⁻³)	(m ⁻¹)
dimethyl sulfoxide	17.62	2520	2.6×10^{-4}
cyclohexanol	7.50	8790	1.7×10^{-4}
anisole	5.05	23000	2.1×10^{-4}

- a area under curve from time t_0 (start of experiment) to time at which absorbance reading is 0.25 of the highest value recorded.
- b for 150 μ L of liquid applied to glass frit. See Table V for temperature at which experiments were run.

Liquid ^a	Ambient Temperature	Volatility @ T°C	(A.U. min)	
Liquid	(°C)	(mg m ⁻³)		
TEP	25.0	2,140	12.80 ± 0.33	
DMSO	25.0	2,520	11.25 ± 0.06	
ACP	23.5	2,934	10.27 ± 0.07	
DMM	22.0	2,983	11.60 ± 0.30	
EMC	23.5	4,963	6.71 ± 0.15	
CHA	25.0	8,790	5.96 ± 0.13	
NAA	25.0	12,600	3.99 ± 0.12	
lAA	25.0	17,600	3.43 ± 0.14	
ANS	21.0	18,590	3.54 ± 0.05	
BTA	24.0	26,410	3.19 ± 0.14	

a see Table I for compound identification.

b area from time t_O until time at which absorbance reading is 0.25 of the highest value recorded.

c data reported as mean ± standard deviation for three replicate runs.

Liquid ^a	Ambient Temperature	Volatility @ T°C	Curve Areabc (A.U. min)	
Liquio	(°C)	(mg m ⁻³)		
TEP	25.0	2,140	19.07 ± 0.56	
DMSO	25.0	2,520	17.62 ± 0.22	
ACP	23.5	2,934	15.61 ± 0.13	
DMM	22.0	2,983	15.79 ± 0.18	
EMC	23.5	4,963	11.27 ± 0.27	
CHA	25.0	8,790	7.50 ± 0.25	
NAA	25.0	12,600	6.39 ± 0.08	
IAA	25.0	17,600	5.21 ± 0.14	
ANS	21.0	18,590	5.05 ± 0.16	
BTA	24.0	26,410	4.72 ± 0.14	

a see Table I for identification.

b area from time t_O until time at which absorbance reading is 0.25 of the highest value recorded.

c data reported as mean ± standard deviation for three replicate runs.

TABLE VI

Volatility — Drying Curve Area Functiona Parameters

Liquid Volume	Function Parameters		Correlation
(μ L)	m	b	Coefficient
150	- 0.596	3.260	0.9966
100	- 0.587	3.053	0.9900

a Log_{10} (Area) = $m log_{10}$ (Volatility) + b

TABLE VII

Comparison of Data for Glass Frit Versus Fabric Substrates

Liquid Volume: $150 \mu L$

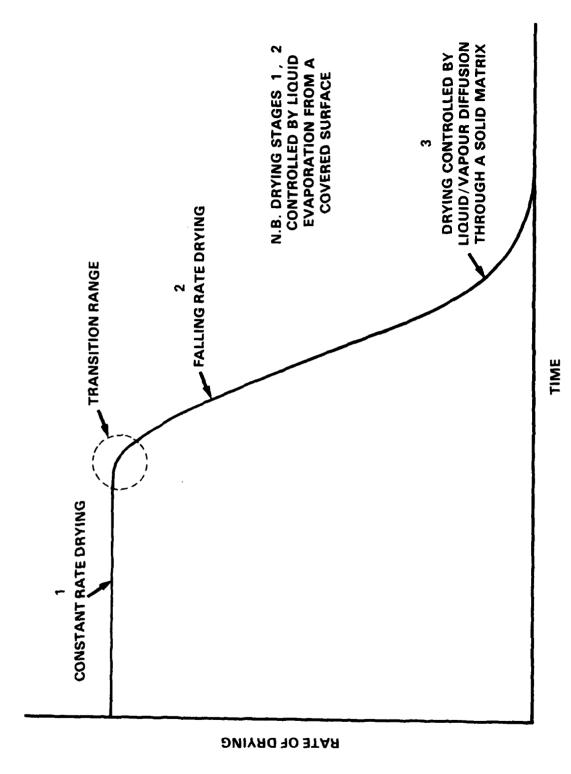
Liquida	Ambient	Curve Areab		Relative
	Temperature (°C)	Fabrice	Glass Fritd	Difference (%)
TEP	25.0	18.62	19.07	- 2.4
DMSO	23.0	19.10	19.05	+ 0.3
ACP	25.0	13.49	14.79	- 9.6
DMM	24.0	14.19	14.76	-4.0
EMC	25.0	10.20	10.96	- 7.5
CHA	25.0	8.02	8.32	- 3.7
NAA	25.0	6.09	6.39	- 4.9
IAA	25.0	5.48	5.43	+ 0.9
ANS	24.5	4.77	4.73	+ 0.8
BTA	24.0	4.45	4.27	+ 4.0

- * see Table 1 for identification.
- area from time to until time at which absorbance reading is 0.25 of the highest value recorded.
- c data reported for a single run in each case.
- area is taken from Figure 4 at the corresponding volatility and temperature T.
- Difference = 100 × Curve Area (FABRIC) Curve Area (FRIT)

 Curve Area (FABRIC)

ANNEX B

LIST OF FIGURES



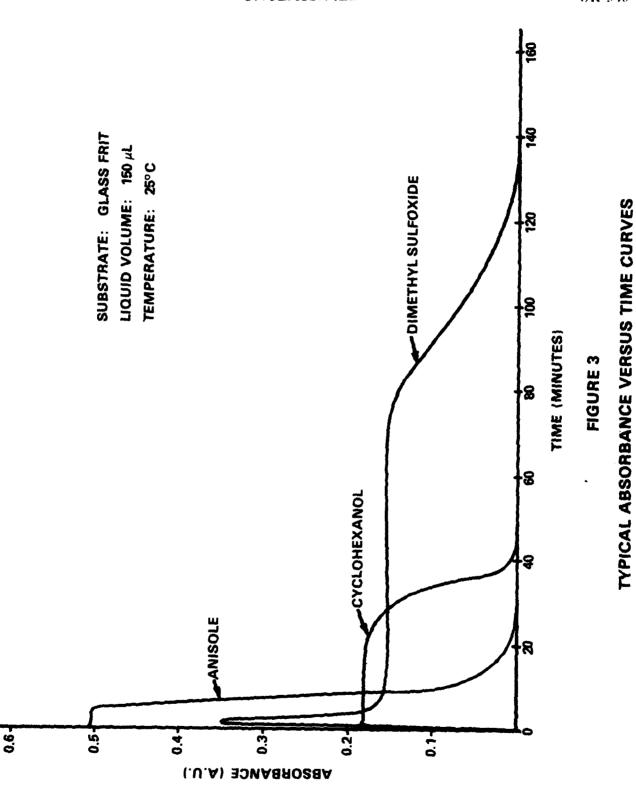
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FIGURE 1

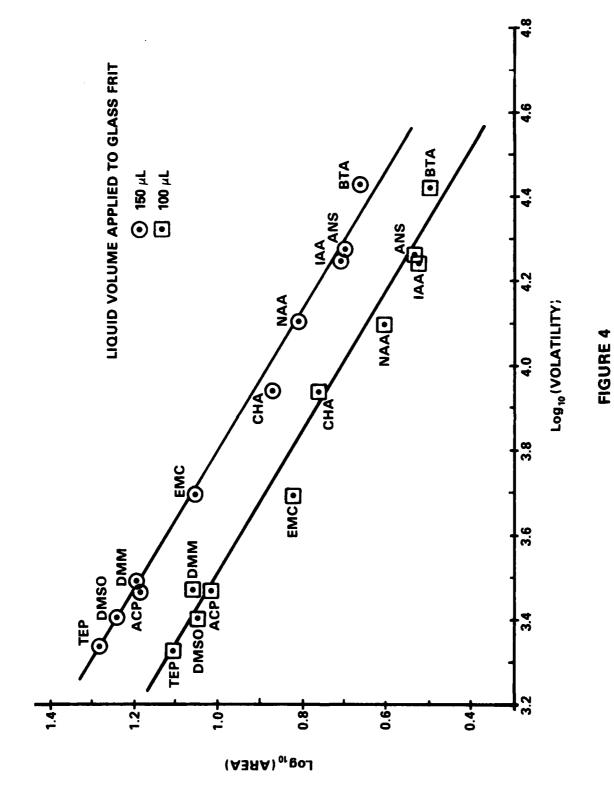
TYPICAL DRYING CURVE FOR A WETTED SOLID UNDER CONSTANT EXTERNAL CONDITIONS

FIGURE 2. APPARATUS FOR MEASI'RING LIQUID VOLATILITY

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RELATIONSHIP BETWEEN VOLATILITY AND DRYING CURVE AREA

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1;	SUPPLEMENTARY NOTES	12. SPONSORII	NG ACTIVITY			
13	A method is described for volatility at ambient temperatures pressure or use a predictive vapour. The method is based on the drying of the dr	without the r pressure-1	e need to m temperature	neasure vapour e relationship.		

controlled conditions and relating the rate of drying to liquid

vapour concentration over time using an infrared gas analyzer.

volatility. The rate of drying is determined by monitoring changes in

1

KEY WORDS

METHODOLOGY
VOLATILITY
LIQUIDS
VAPOUR
DRYING
INFRARED ANALYZER

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